Oct 1, 2003

Mr. Stephen L. Frankiewicz
EG&G Defense Materials, Inc.

Mr. Thaddeus Ryba, Jr.
TOCDF Deputy Site Project Manager
Department of the Army
US Army Chemical Materials Agency
11620 Stark Road
Tooele, UT 84074

Dear Messrs. Frankiewicz and Ryba:

In your letter dated September 3, 2003, you propose several action items in preparation for conducting additional tests in the wake of your review of results from the RCRA Trial Burn of July 2003 for VX M55 Rockets. Trial Burn results revealed that Tooele Chemical Disposal Facility (TOCDF) did not achieve the 99.9999% destruction and removal efficiency (six 9s DRE) required by rule of PCB incinerators. TOCDF concluded that portions of the PCBs found in samples collected during the Trial Burn did not originate from the incinerator. The National Program Chemicals Division (NPCD) of the U.S. Environmental Protection Agency (EPA) reviewed the Trial Burn data and agrees with TOCDF's conclusion. Furthermore, TOCDF also suggested an alternative method for calculating the PCB DRE. TOCDF proposed to subtract the quantity of PCBs found in the Field Blank sample from the quantity of PCBs in Trial Burn stack samples. TOCDF contends that the Field Blank sample represents the background PCB contamination in the emission samples and should be subtracted from the emission, thus allowing the six 9s DRE standard to be apparently met. NPCD disagrees with this approach. Although characteristics of the Field Blank closely resemble those from the other emission samples, NPCD believes that the source of the PCBs in the Field Blank is related to PCBs in the stack emissions but does not originate from the gas in the stack.

TOCDF Proposed Supplementary Tests and Procedures

TOCDF has proposed the following actions in supplementary tests to confirm that the six 9s can be achieved:

- Conducting a 72-hour shakedown of the incinerator with rocket feed rates approaching those of the July trial burn.

CONCURRENCES

SYMBOL		7407T					
SURNAME		DOD					
DATE		10/1/03					

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OCT - 1 2003

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

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TOCDF Proposed Supplementary Tests and Procedures

TOCDF has proposed the following actions in supplementary tests to confirm that the six 9s can be achieved:

- Conducting a 72-hour shakedown of the incinerator with rocket feed rates approaching those of the July trial burn;

- During the shakedown collecting PCB emission samples as recommended by EPA OPPT (As part of the continuing discussion on this matter, OPPT (NPCD) emailed a draft response letter replying to TOCDF's draft of this letter. In the draft response, NPCD outlined procedures recommended for resumption of operations);

- Initiating sampling for PCB emissions after steady-state feed rate is achieved; and,

- Following the operative provisions of either the National Permit or the Demonstration Permit issued April 19, 2002 after sampling is initiated.

In general, NPCD agrees with the procedures proposed by TOCDF which lead to the second round of tests suggested by TOCDF. However, NPCD recommends several additional steps to be implemented prior to initiating the tests.

NPCD Recommended Supplementary Tests and Procedures

NPCD recommends that the incinerator be inspected for PCB contamination prior to shakedown operations. After the July, 2003 Trial Burn, TOCDF curtailed incinerator operations upon discovering that the PCB DRE requirement was not achieved. Subsequently, tests were not performed to verify whether the contaminating PCBs were eliminated from the incinerator. To ensure that PCB contaminants do not interfere with the additional testing, NPCD recommends the following actions by TOCDF.

- Conduct a Fuel-only/Baseline (FOB) emission monitoring for PCBs prior to initiating the 72-hour shakedown; results of this sample should ensure that the contaminant PCBs have been purged from the incinerator. Two four-hour-minimum stack samples should be collected; one following the sample train preparation procedure used to collect samples during the July, 2003 Trial Burn (assumed to be Method 23A), and the second using the laboratory cleanup procedure at EPA SOP MSL-M-090-00 (Appendix B).

- Implement a 72-hour shakedown period with "ramping up" of rocket feed rates to approach those of the July Trial Burn. (TOCDF must ensure that enough M55 Rockets remain to complete the series of tests and the trial burn as outlined in the **Miniburn and Trial Burn** section below.)

- Review the FOB results to ensure that contaminant PCBs were eliminated, followed by collecting of PCB emission samples as recommended by NPCD (the series of tests are detailed below) when steady state-feed rate is achieved.

- Operate the incinerator following provisions of the Demonstration Permit issued April 19, 2002, which require demonstrating the achievement of the six 9s DRE during the **miniburn** prior to proceeding with the Trial Burn.

Miniburns and Trial Burn

NPCD believes that a series of "miniburns" must be performed focusing on demonstrating the capability of the incinerator to achieve the six 9s DRE and simultaneously determining the source of the contaminant PCB. On completion of the miniburns, TOCDF should implement a trial burn to confirm the findings. NPCD recommends the procedures below to be followed, i.e., two miniburns followed by a Fuel-only/Baseline test in the following sequences. Analysis of the samples shall be limited only to PCBs because this series of tests must be expedited to confirm the incinerator's capability to achieve six 9s DRE as well as to determine the source of the contaminant PCB emissions.

Miniburn 1 (M1)	One four-hour sampling at a feed rate of 21-22 rockets per hour. The sampling train shall be prepared in a manner identical to the July 2003 Trial Burn with the exception of the XAD-2 resin and glass fiber filters. The XAD-2 resin shall be new and cleaned, as well as the glass fiber filters using the EPA SOP MSL-M-090-00.
Miniburn 2 (M2)	One four-hour sampling at 21-22 rockets per hour. The sampling train shall be prepared in a manner identical to the July 2003 Trial Burn preparing the XAD-2 resin and the glass fiber filters identical to the July 2003 Trial Burn.

Two field blanks, the first using the sampling train described in test M1 and a second using the sampling train described in M2, shall be prepared and set up during the Miniburn tests. Prior to performance of the mini-burn, TOCDF shall provide NPCD with the procedures employed in preparation of the sampling trains used in the July 2003 Trial Burn.

For the trial burn, NPCD recommends the following procedure.

Trialburn 1 (T1)	One four-hour Fuel-Only/Baseline test, using procedures in the July 2003 Trial Burn, if judged to be adequate by review of the Miniburn test results.
Trialburn 2 (T2)	Four four-hour sampling at 21-22 rockets per hour, using procedures in the July 2003 Trial Burn, if judged to be adequate by review of the Miniburn test results.
Trialburn 3 (T3)	If the Miniburn test results indicate that lab procedures for preparing the XAD resin and/or the sampling train preparation procedures are inadequate, then the Trial Burn shall be scheduled to accommodate cleaning procedures incorporated in EPA SOP MSL-M-090-00.

Interim Operations (continuation of operations to incinerate remaining rockets after this Trial Burn) will depend on whether results from the Miniburn tests indicate that six 9s DRE were achieved, but otherwise will follow the terms set forth in the TSCA Demonstration Permit issued April 19, 2002.

Alternative DRE Calculation

TOCDF claims that PCBs from a source not related to the incinerator contributed to the PCB emissions during the Trial Burn. TOCDF further claims that this contaminant PCB is the background material caught in the Field Blank sample and therefore, the quantity of PCB in the Field Blank sample should be subtracted from the stack emission samples thus reflecting the actual PCB emissions. With the Field Blank value subtracted, the stack PCB emission is reduced enough to meet the six 9s DRE requirement. NPCD believes this is inappropriate, and concludes that the Field Blank material did not originate from stack gas and is not background contamination.

Initially, the Field Blank sampling train is a passive equipment assembly - stationary throughout the series of tests. Consequently, NPCD believes that such a small quantity of gas flowing through the Field Blank is insignificant when compared to the approximately 150 cubic feet of gas flowing through the stack samples. The only period in which gas may enter the Field Blank sample is during the leak check at the end of a test. This minor quantity of gas could not have been the source of the 58 ng. of PCB found in the Field Blank sample. The 58 ng. is within the range of PCB found in all the stack samples (33 to 114 ng.), including those from the dioxin and hazardous pollutant samples. Because a small quantity of gas passing through the Field Blank could not have contributed such a large quantity of PCB, NPCD concludes that the PCB in the Field Blank sample more likely originated at a site remote from the incinerator.

Analysis of chromatograms from all the PCB related samples reveals patterns which are very similar, indicating high probability that the contamination originated from a single source (see Appendix A, Figures 1, 2 and 3). TOCDF indicates that the swipe sample taken where the Field Blank sample was collected and the ambient air sample collected in the stack sampling enclosure contain low levels of PCB (4.8 ng. and 4.5 ng. respectively). Therefore, TOCDF concludes that the source of the contamination was not within the sampling enclosure. NPCD disagrees with TOCDF's conclusion. Close examination of the chromatograms from the swipe and ambient air sample reveal patterns closely resembling those from the other samples (see Appendix A). The high molecular weight patterns (Figure 3) reveal some variance among the samples, but the characteristics are similar, indicating the contamination may be from a common source. However, that source has not been determined. Therefore, NPCD cannot concur with the proposed alternative DRE calculation, i.e., subtraction of the quantity of PCB in the Field Blank sample from those in the stack emission samples. If you do not agree to operate in compliance with EPA's recommended supplementary tests and procedures, you must submit a modified plan to determine the source of the PCB contamination and to collect additional monitoring data during any subsequent burn.

Please direct questions regarding this letter to Hiroshi Dodohara of my staff at 202-566-0507.

Sincerely,

Maria J. Doa

Maria J. Doa, Ph.D.
Acting Director
National Program Chemicals Division

cc. Dan Bench
US EPA, Region 8

Craig Brown
US EPA, Region 4

Jim Sales/Lou Robert
US EPA, Region 6

Dan Duncan/Catherine Massimino
US EPA, Region 10

Joe Stang
US Army CMA

Dennis Hooton
MRI

APPENDIX A

Chromatograms of Samples from July 2003 Trial Burn at the Tooele Chemical Disposal Facility

Sample Description:

Field Blank

PCBs Introduced into Incinerator. A quality control/quality assurance sample collected in a stack sampling train assembly, but stationary and passive, placed near the location of the stack sampling equipment in the stack sampling enclosure. Stack gas is not passed through the Field Blank.

Fuel-Only/Baseline

No PCBs Introduced into Incinerator. The initial test run during the July '03 Trial Burn where no waste feed was introduced into the incinerator. Only clean fuel was used.

Run 2

PCB Introduced into Incinerator. Test Run 2 of the Trial Burn during which Mk 55 VX Rockets with PCB-contaminated shipping/firing tubes were fed to the incinerator.

Run 6

No PCB Introduced into Incinerator. Test Run 6 of the Trial Burn during which Mk 56 Warheads with shipping/firing tubes containing no PCBs were fed to the incinerator.

Ambient Air

Incinerator Idle. Ambient air sampled in the stack sampling enclosure about one month after the Trial Burn was completed.

Swipe

Incinerator Idle. Swipe sample taken near the site where the Field Blanks were located.

Figure 1. Chromatograms in the Retention Time Range 18 min. to 22 min.

All samples display virtually identical chromatographic pattern with only insignificant differences.

Figure 2. Chromatograms in the Retention Time Range 23 min. to 33 min.

All samples display virtually identical chromatographic pattern with only insignificant differences.

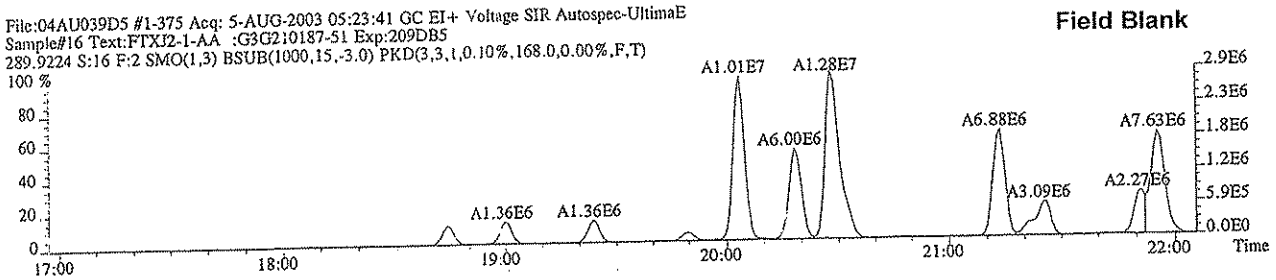
Figure 3. Chromatograms in the Retention Time Range 40 min. to 45 min.

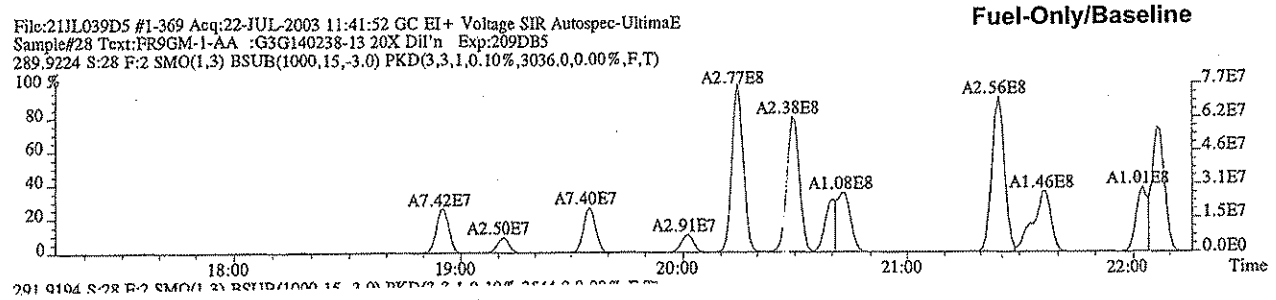
With the exception of the Ambient Air sample, all have similar chromatographic patterns, Several of the peaks in the Ambient Air samples do not display the prominence that other samples do. This retention time window displays organic compounds of higher molecular weights.

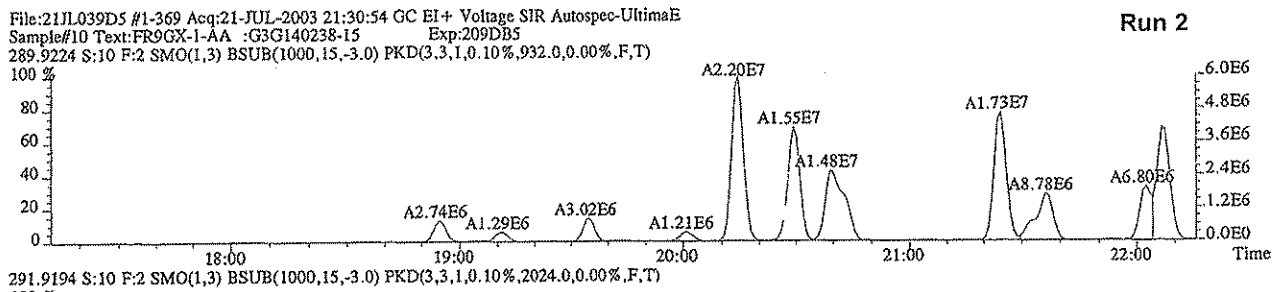
Appendix B

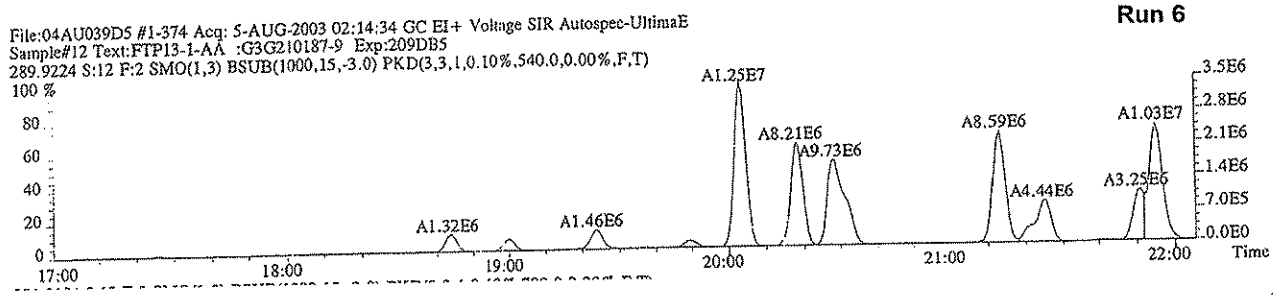
Standard Operating Procedure MSL-M-90-00
HOC Sampling Media Preparation and Handling:
XAD-2 Resin and GF/F Filters

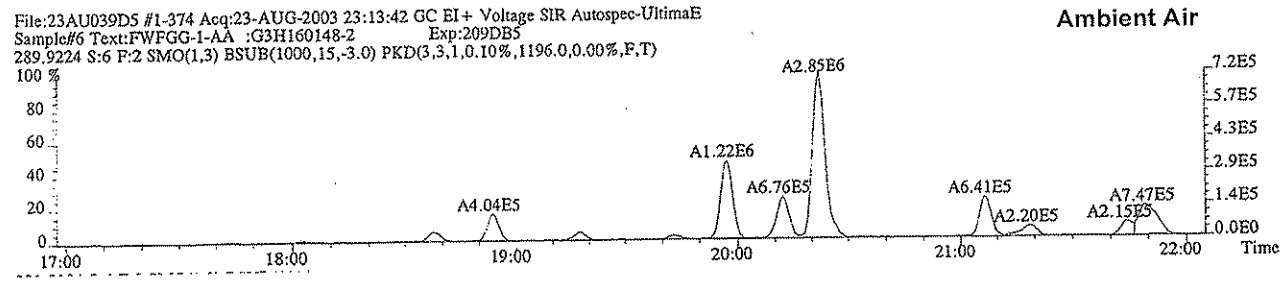
Figure 1. Chromatograms in the Retention Time Range 18 min. to 22 min.











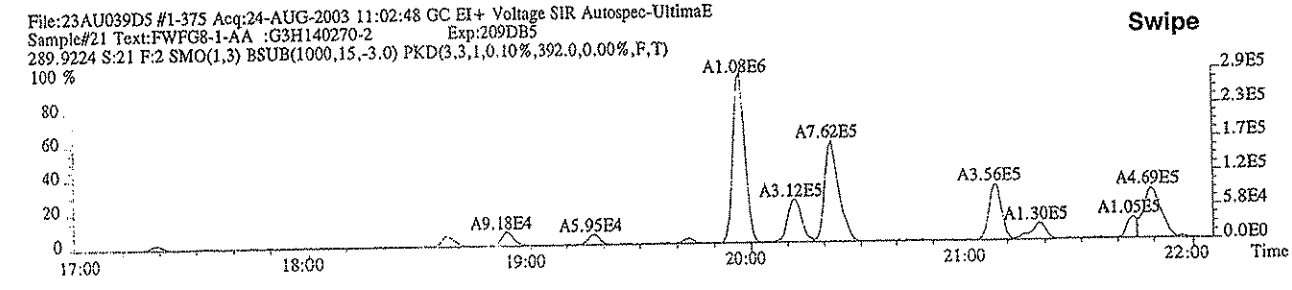
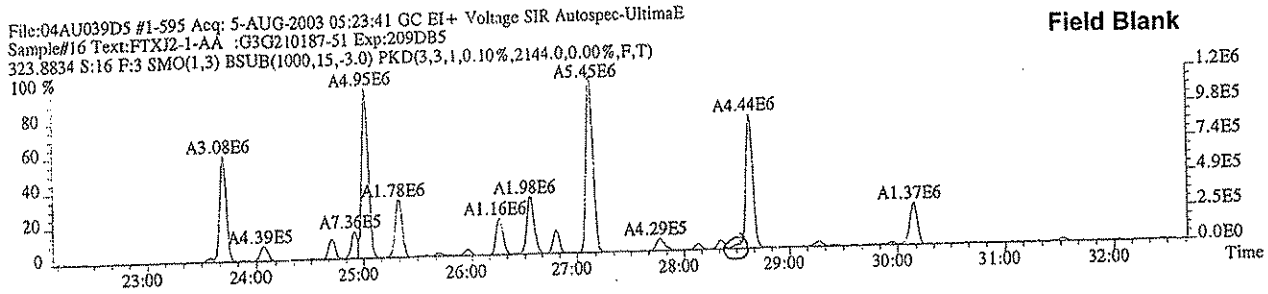
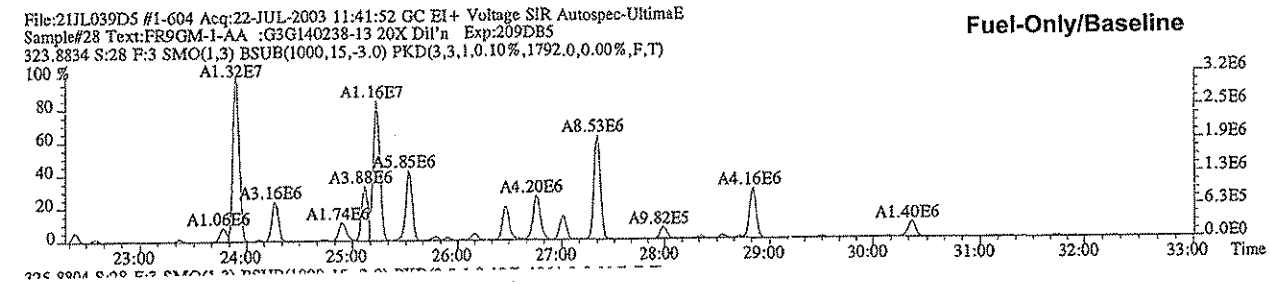
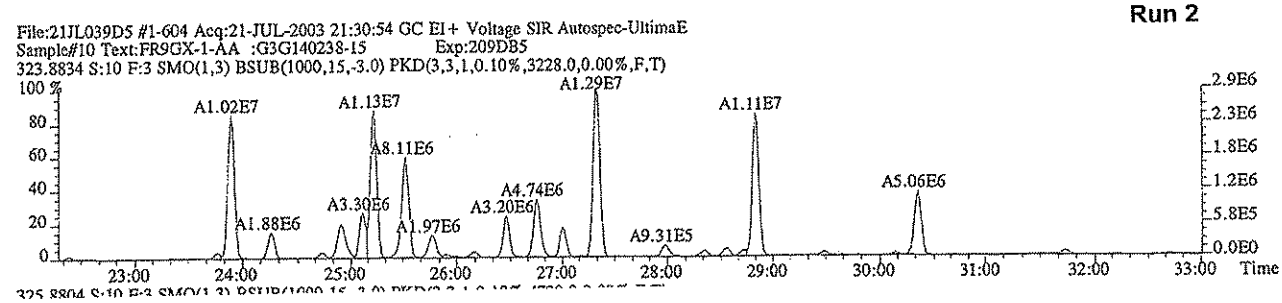
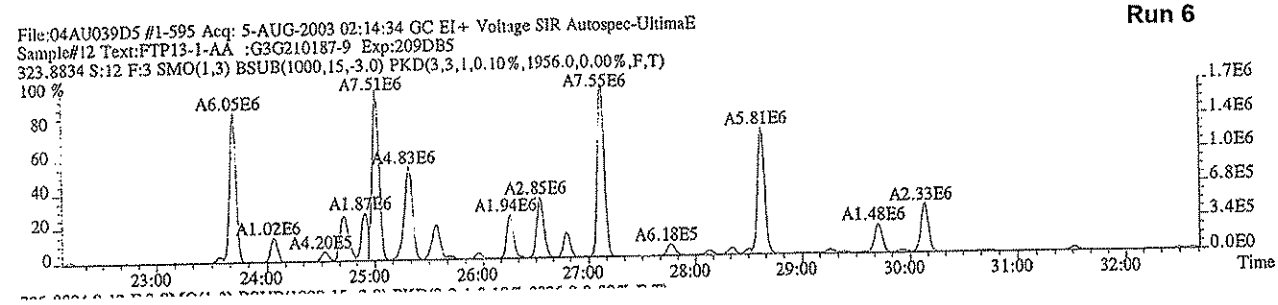


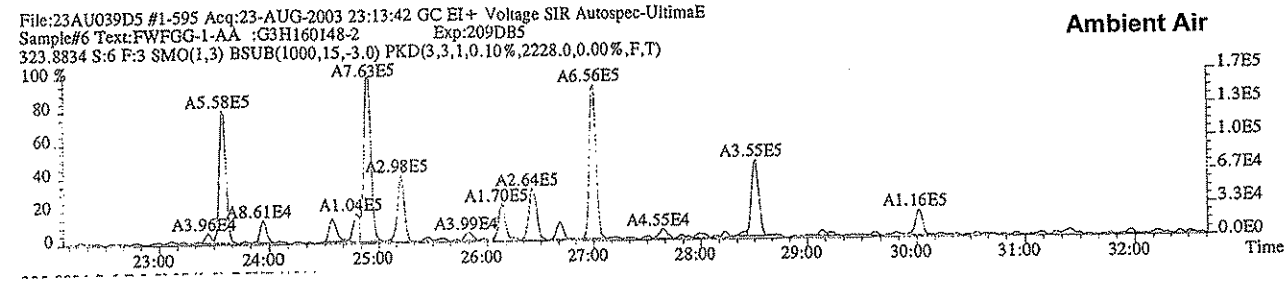
Figure 2. Chromatograms in the Retention Time Range 23 min. to 33 min.











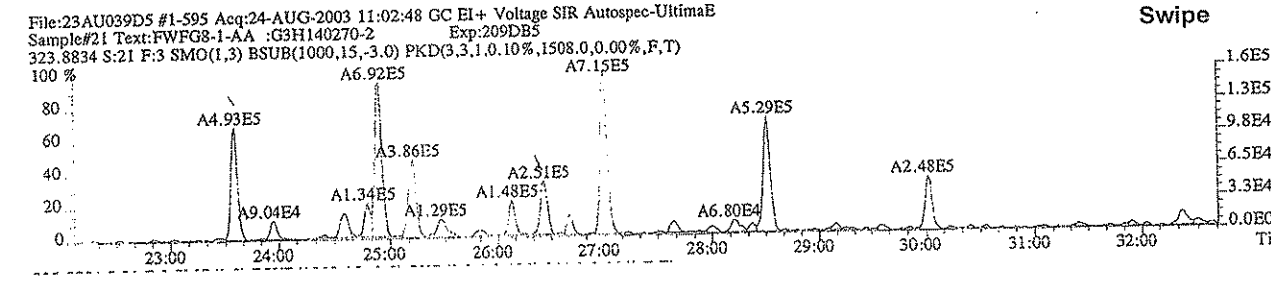
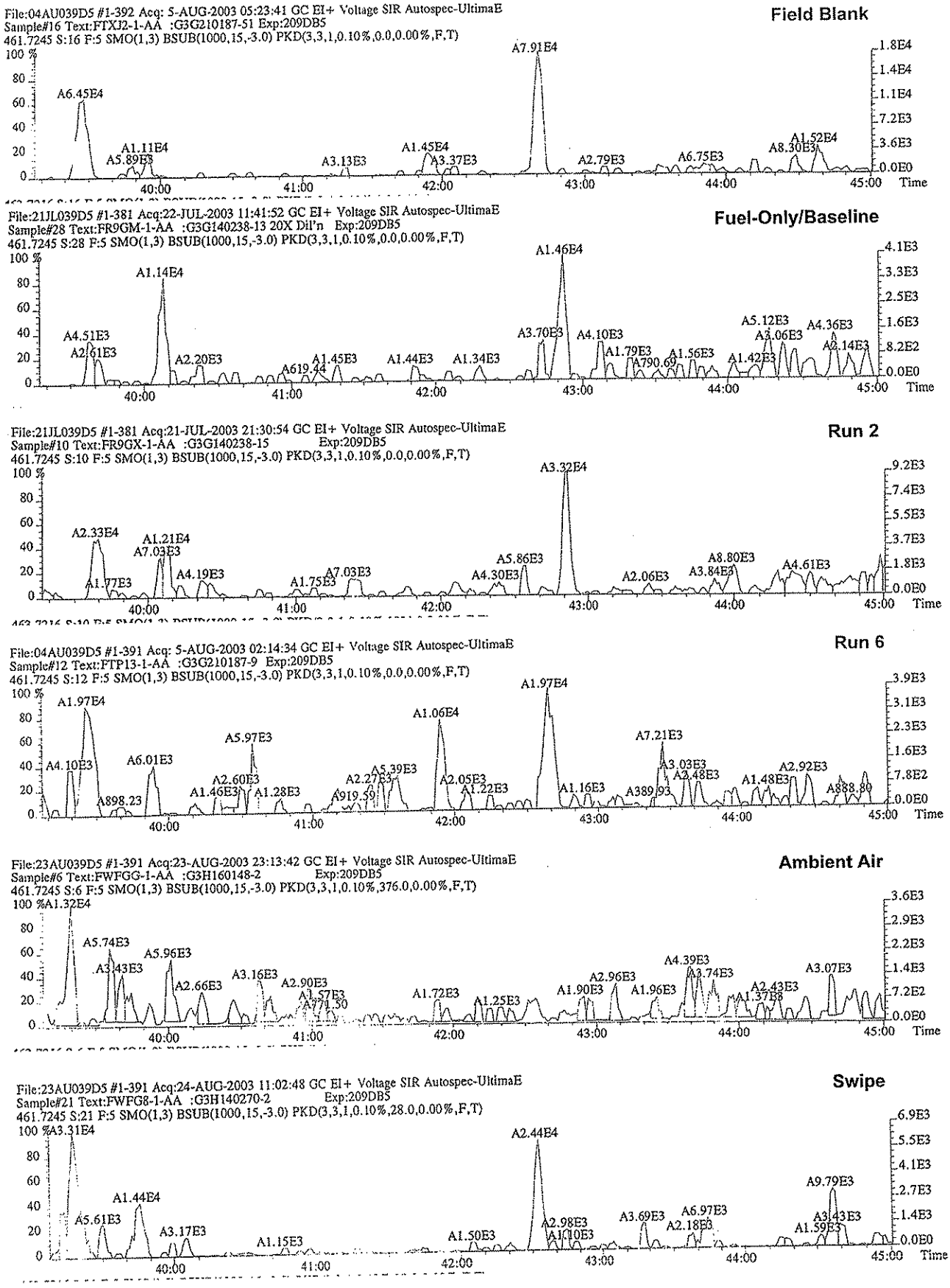


Figure 3. Chromatograms in Retention Time 40 min. to 45 min.



HOC Sampling Media Preparation and Handling; XAD-2 Resin and GF/F Filters

Eric Crecelius and Lisa Lefkovitz
Pacific Northwest National Laboratory
Battelle Marine Sciences Laboratory
1529 West Sequim Bay Road
Sequim, WA 98382

Standard Operating Procedure MSL-M-090-00

November 1994

HOC Sampling Media Preparation and Handling; XAD-2 Resin and GF/F Filters

1.0 Scope and Application

This method is applicable to the preparation of sampling media used in the collection of hydrophobic organic compounds (HOCs) from water.

The dissolved HOC phase is collected on XAD-2 resin, a macroreticular resin bead that selectively scavenges HOC from other media such as water and/or air. The manufacturing process of this material results in very dirty product and a very rigorous clean-up procedure is needed to remove these potential interferences. Also, care needs to be taken when handling the resin to avoid damage of the beads which could lead to reintroduction of the original contaminants possibly bound into the beads.

Glass fiber filters are used to filter out the "particulate" fraction of the water. Since HOCs are preferentially bound to particulates in these media, this material needs to be isolated to determine the particulate-bound fraction of HOCs present. Again, special cleaning and handling procedures are required to obtain filters clean enough for trace level HOC analyses.

2.0 Definitions

HOC	Hydrophobic organic contaminants
GF/F	Glass fiber filter
XAD-2	Manufacturers name for a class of polymeric resin beads used to isolate HOCs from water.
LRB	Laboratory Record Book

3.0 Responsible Staff

Laboratory Supervisor. A Technical Specialist or Scientist having expertise in the principles involved with this procedure and in the use of laboratory operations in general. Responsible for ensuring that analysts are trained in the use of the instrument and that maintenance logs are being completed.

Analyst. A Technician, Technical Specialist, or Scientist assigned to utilize the instrument for actual sample analysis using this procedure. Responsible for 1) understanding the proper use of tools and solvents; 2) recording information regarding maintenance of the instrument in the appropriate logbooks; 3) reporting any significant problems with the instrument to the Laboratory Supervisor; and 4) tabulating and reporting sample data to the Laboratory Supervisor.

4.0 Procedure

4.1 XAD-2 Resin

XAD-2 resin can be obtained from a number of different vendors but is manufactured solely by Rohm and Haas. The size of the resin beads is 20-60 mesh. A rigorous clean-up procedure must be applied prior to use of the resin for collection of HOCs.

4.1.1 Apparatus and Reagents

Methylene Chloride, Acetone, Hexane, Methanol; HPLC grade or better
Glass wool/soxhlet extracted in hexane/acetone (50:50)
Amberlite XAD-2 Resin, 20-60 mesh. Rohm and Haas manufacturer

4.1.2 Resin Clean-up Method

The XAD-2 resin is cleaned in the lab by a series of solvent extractions in a large soxhlet apparatus (or in multiple set-ups). The resin is extracted sequentially for 24 hours each in methanol, acetone, hexane and methylene chloride. This is followed by sequential 4-hour extractions in hexane, acetone and methanol which cycles the resin back to a polar solvent. The methanol is then displaced from the resin by numerous rinses with organic-free water. The resin can be stored at this point in clean jars immersed in the water in a dark place for up to three months. The final four-hour hexane extract may be used for a laboratory XAD-2 blank. The last methanol rinse may be used as the starter methanol on the next XAD-2 batch.

4.1.3 QC of Resin/Is it Clean?

A portion of the resin from each clean-up batch must be tested to ensure a thorough clean-up has been performed. As noted above, the final four-hour hexane extract may be used for a laboratory XAD-2 blank. Alternatively, a representative amount of pre-cleaned resin from a given clean-up batch may be extracted using the extraction scheme to be used for the project of interest and the extract analyzed as a resin blank. The cleanliness of the resin will be evaluated on a project specific basis.

4.1.4 XAD-2 Resin Column Preparation

XAD-2 resin must be packed into a column for use as a sampling media for dissolved phase HOCs. The resin columns may be glass, stainless steel or teflon and can vary in size. This procedure is specific to glass columns with dimensions of 300 mm x 50 mm, fitted with nylon end plugs sealed with viton O-rings.

XAD-2 resin columns are prepared by first attaching one teflon adaptor with a swagelock fitting and a 3 inch length of latex tubing to one end of the glass column, and pushing a large plug of cleaned glass wool into the bottom. The column is filled about ½ full with organic free water and clean resin is poured into the column in a slurry to a final packed length of ~19.5 cm (~400 cc). The resin is packed by pumping excess water out from the bottom using a water aspirator peristaltic pump but always maintaining enough water in

the colimn to cover the resin. The column should not contain air bubbles or channels. Glass wool is added at the top to take up the space between the XAD-2 and the column threads. A solid nylon end cap with O-ring is placed on the top and then, after inverting the column and unscrewing the adaptor, the other end is capped in the same fashion.

4.1.5 Column Handling and Storage

Upon receipt of a cleaned batch of resin, the batch is named for the date of receipt and recorded in the project LRB. A copy of the chromatogram of the resulting XAD-2 resin blank that is determined for that batch is also included in the LRB. All columns are assigned individual numbers based on the resin batch number which is written in permanent marker on a piece of tape wrapped around the outside of the column. Columns are stored in a clean, cool place in the dark and can be stored up to 6 months prior to use. After sampling, columns should be stored at 4°C in the dark. There is no holding time for sampled resin columns prior to extraction.

4.2 Glass Fiber Filter

4.2.1 Apparatus and Reagents

Muffle Furnace
Al foil, heavy duty, extra wide
Whatman 293 mm GF/F 0.7µm nominal pore size glass fiber filters

4.2.2 Filter Clean-up Method

Filters are wrapped in a single layer of heavy duty aluminum foil which is sealed around the filter to create a "bag." The filter and aluminum foil are then ashed for four hours at 450°C (±20°C).

4.2.3 QC of Filter/Is it Clean?

One filter (or more, since more than a single filter may be used for a given sample) should be extracted using the extraction scheme to be used for the project of interest and the extract analyzed as a filter blank. The cleanliness of the filter will be evaluated on a project specific basis.

4.2.4 Filter Storage and Handling

Cleaned filters are stored inside of their foil bags in a clean, cool place prior to sampling. Multiple filter/ foil units can be stored in sealed polyethylene bags for storage and/or shipping. The bags containing cleaned filters from the same lot are labeled as the preparation date of filters, the initials of the technician who prepped them, the number of filters in the bag and the page number of the LRB where the preparation information is recorded.

After filters are used for sampling, they are to be folded in quarters (pie shaped) and placed in sealed ashed foil bags and stored frozen in plastic bags. There is no holding

time for storage of sampled filters prior to extraction.

4.3 Interferences

Take appropriate precautions to prevent contamination of any equipment associated with this analysis.

5.0 Data Analysis and Calculations

Not applicable.

6.0 Quality Control

6.1 Solvent Blanks. Use only HPLC grade or higher purity solvents for clean-up. Only a single lot number of each solvent should be used. A solvent blank test will be performed upon the start of a new lot number by concentrating a representative volume of solvent to 1 mL and analyzing on the appropriate analytical instrument. Cleanliness of the solvent will be determined on a project specific basis.

6.2 Resin Blank per batch. Resin used for a given project should be isolated to a single manufacturer's lot number since the original level of contamination of the resin can vary significantly with lot. Resin blanks will be analyzed per clean-up batch as specified in Section 4.1.3. Cleanliness of the resin will be determined for each new lot number on a project specific basis.

6.3 One Filter blank per batch. Filters used for a given project should be isolated to a single manufacturers lot number. Filter blanks will be analyzed per clean-up batch as specified in Section 4.2.3. Cleanliness of the filters will be determined on a project specific basis.

6.4 All results will be recorded in an LRB which is reviewed periodically by the laboratory supervisor and monthly by the project manager.

7.0 Safety

All analysts following this procedure should be aware of routine laboratory safety concerns, including the following:

7.1 Protective clothing and eyeglasses should be worn when appropriate.

7.2 Proper care must be exercised when processing samples because volatile and flammable solvents are involved.

8.0 Training Requirements

All staff preparing sampling media described above must first read this SOP and then demonstrate proficiency in the process prior to performing the work under the supervision of the laboratory manager.

9.0 References

MSL-A-006. Marine Sciences Laboratory Training.